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COMPOSITES THEORY AND PRACTICE

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COMPARATIVE ANALYSIS OF FAILURE OF Al/GFRP LAMINATES AFTER TENSILE STRENGTH TEST

Fibre-metal laminates are modern composite materials that are replacing certain metal elements in aircraft structures. Such hybrid materials have synergic properties determined by their component properties and configuration. This article presents studies of GLARE laminates consisting of aluminium and glass-epoxy composite sheets manufactured using the autoclave method. 2024T3 aluminium alloy sheets were subjected to chromic acid anodising (CAA) and sulphuric acid anodising (SAA). Three different lay-up configurations of the composite layers were used in the structure of 2/1 laminates: [0°], [0/90°] and [±45°]. Tensile strength studies were conducted using a strength testing machine (MTS 322) in accordance with the ASTM standard for composite materials. Microstructural and fractographic observations were conducted using an optical microscope and a scanning electron microscope (Zeiss Ultra Plus, NovaNanoSEM 450). The tensile strength test did not result in cracking of the metal plies; it was the composite that underwent degradation. After the test, the samples were found to have undergone deformation and delamination as a result of the tension; the laminates with an SAA layer were more greatly affected. The plastic range properties are determined by the fibre configuration. The metal/composite adhesion force was higher than the cohesive force in the composite for all the configurations. The degradation mechanism of the laminate structure during uniaxial tensile strength tests does not depend on the type of anodised layer. The configuration of the fibrous composite layers affects the propagation of cracks in the composite area. Transverse cracking of the fibres, cracking of the anodised layer and decohesion of the matrix with tearing-out of fibres were observed in all the cases. The surface morphology of the fracture caused by the decohesion of the composite in an FML is of the same nature as the fracture in the composite material.

Keywords: **fibre metal laminates, tensile strength, fractography, failure**

ANALIZA PORÓWNAWCZA ZNISZCZENIA LAMINATÓW Al/GFRP W PRÓBIE WYTRZYMAŁOŚCI NA ROZCIĄGANIE

Laminaty metalowo-włókniste to współczesne materiały złożone zastępujące niektóre elementy metalowe w konstrukcjach lotniczych. Taki materiał hybrydowy posiada właściwości synergiczne, determinowane właściwościami komponentów i ich konfiguracją. W niniejszej pracy przedstawiono badania laminatów typu Glare, składających się z blach aluminium i kompozytu epoksydowo-szklanego, wytworzonych metodą autoklawową. Blachy ze stopu aluminium gat. 2024T3 anodowano w roztworze kwasu chromowego (CAA) oraz w roztworze kwasu siarkowego (SAA). W budowie laminatów 2/1 wykorzystane zostały trzy różne konfiguracje ułożenia warstw kompozytu: [0°], [0/90°] oraz [±45°]. Badania wytrzymałości na rozciąganie zostały przeprowadzone przy użyciu maszyny wytrzymałościowej (MTS 322) zgodnie z normą ASTM dla materiałów kompozytowych. Obserwacje mikrostrukturalne i fraktograficzne zostały przeprowadzone przy użyciu mikroskopu optycznego i skaningowej mikroskopii elektronowej (Zeiss Ultra Plus, NovaNanoSEM 450). W teście rozciągania nie nastąpiło pęknięcie warstw metalu, degradacji uległ kompozyt. Po zakończeniu testu wystąpiła deformacja próbek i ich rozwarstwienie na skutek naprężenia, silniejszy efekt zaobserwowano w laminatach z warstwą SAA. O właściwościach w zakresie plastycznym decyduje konfiguracja włókien. We wszystkich układach siła adhezji metal/kompozyt przewyższała siłę kohezji w kompozycie. Mechanizm degradacji struktury laminatów podczas jednoosiowego rozciągania nie zależy od typu warstwy anodowej. Konfiguracja warstw kompozytu włóknistego wpływa na propagację pęknięć w obszarze kompozytu. We wszystkich przypadkach nastąpiło pękanie poprzeczne włókien, pękanie warstwy anodowej oraz dekohezja osnowy z odrywaniem włókien. Morfologia powierzchni przełomu powstałego w wyniku dekohezji kompozytu w FML ma taki sam charakter jak przełom w materiale kompozytowym.

Słowa kluczowe: **laminaty metalowo-włókniste, rozciąganie, fraktografia, zniszczenie**

INTRODUCTION

In recent years, composite structures have become highly popular materials for different applications, predominantly in the aerospace industry. Their large spectrum of properties, such as strength and stiffness-to-weight ratio, fatigue characteristics and corrosion resistance comprise several highly distinctive advantages for applications in lightweight primary structures. The next generation of hybrid composites used in the aircraft industry are fibre-metal laminates. Fibre metal laminates (FML) are advanced hybrid materials composed of alternating layers of metal and composite with a polymer matrix reinforced with continuous fibre [1]. FMLs were introduced in the aerospace technology at the turn of the 21st Century. The first laminates applied and studied to date are GLARE® [1, 2]. GLARE is composed of aluminium alloy sheet layers and composite layers featuring an epoxy matrix reinforced by continuous unidirectional glass fibre. Such hybrid materials have synergic properties determined by the component properties and configuration. [3]. The alternating layers of metal and composite facilitate the manufacturing of a material with the desired mechanical properties. The alloy grade, sheet thickness and number of layers (2/1, 3/2 etc. laminates) are selected for the metal layers. For the composite layers, the selected aspects include the grades of the polymer and fibre, the direction of the layers of unidirectional tape or the weave type. This leads to obtaining very high mechanical properties in relation to material density (e.g. tensile strength, fatigue strength, impact resistance) [1, 2, 4].

The procedures for designing and testing are predominately based on experimentally obtained data and analytical and numerical models [3-5]. Tension, compression, shear, bending, fatigue, resistance to impact are the main tests used to define FML mechanical properties [1, 5-7].

The mechanical properties of composite materials are governed by the adhesion between the fibre and the matrix. Beside this, the same properties of FMLs are governed by the interface bond between the composite layer and the metal layer. The adhesion force depends primarily on the aluminium surface treatment method. One of the basic methods of aluminium surface treatment is anodising. In industrial environments, the anodising process has for years involved chromic acid (CAA), which provides excellent protection against corrosion and good metal-metal and metal-polymer adhesive bonds. Due to the harmfulness of Cr(VI) compounds and environmental considerations, this electrolyte is being replaced with solutions of other acids, particularly by US producers. Sulphuric acid anodising (SAA), phosphoric acid anodising (BAA), boric-sulphuric acid anodising (BSAA) and other anodising methods are used [3, 4].

Most studies of the properties of GLARE-type FMLs concern materials with a CAA layer and an additional thin layer of primer. With the recommendation to phase out CAA it has become necessary to determine the durability of the aluminium-composite bond and the mechanism of its degradation after using other anodising solutions.

The tests presented in this article use two aluminium sheet anodising methods: chromic acid anodising (CAA) and sulphuric acid anodising (SAA). Al/GFRP laminates with different fibre configurations were subjected to a tensile strength test in order to compare the tensile strength and structure degradation mechanism in manufactured GLARE-type laminates.

MATERIALS AND METHODS

The subject of the study comprised 2/1 fibre metal laminates based on aluminium sheets. The inner layer was a polymer composite based on glass fibres. The individual components of the laminate were:
- aluminium alloy EN AW-2024T3 sheets with a thickness of 0.5 mm,
- unidirectional prepreg tape glass fibre-epoxy resin TVR 380 M12/26%/R-glass (Hexcel, USA) with a thickness of 0.255 mm; the nominal relative volume of the fibres was about 60%.

The aluminium sheets were anodised in chromic acid (CAA) and sulphuric acid (SAA) in an industrial anodising process according to procedures for aviation. A thin layer of primer containing a corrosion inhibitor was applied immediately after anodising.

The laminates were manufactured in the laboratory of the Department of Materials Engineering at the Lublin University of Technology. The autoclave method was used (Fig. 1).

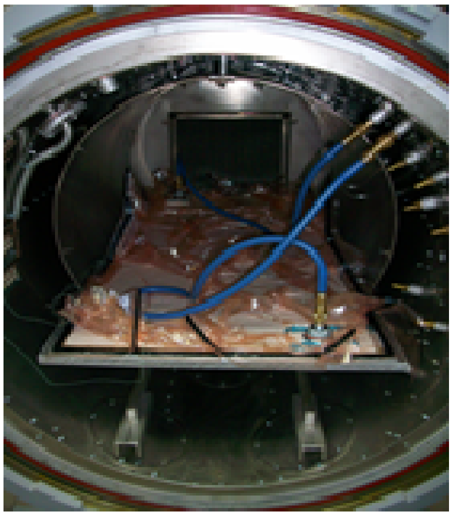

Fig. 1. Autoclave chamber with prepared feed, Scholz Maschinenbau, Germany; Department of Materials Engineering Laboratory in LUT

Rys. 1. Komora autoklawu z przygotowanym wsadem, Scholz Maschinenbau, Germany; Laboratorium Katedry Inżynierii Materiałowej, PL

The curing process parameters of the FMLs were as follows: pressure 0.45 MPa, vacuum −0.08 MPa, curing temperature 403 K, speed of heating and cooling 0.033 K/s.

Fibre metal laminates were manufactured in different configurations of fibre orientation: Al/GFRP [0°], Al/GFRP [0/90°], Al/GFRP[±45°].

The laminate thickness was 1.5 mm; the thickness of the composite layer was equal to the thickness of the metal layer (0.5 mm).

The FML tensile strength tests were modelled on the standard for layered composites as there are no standards designed specifically for testing this group of materials.

The tensile strength test was performed with the use of a universal testing machine (MTS 322). An axial extensometer (Epsilon and MTS) with a 20÷50 mm measurement base and a transverse one (depending on the width of the specimens) were used (Fig. 2) in the test. The crosshead rate was 2 mm/min. The dimensions of the specimens were 150x15x1.5 mm for [0°] and 150x20x1.5 mm for other stacking sequences of polymer composites in FML. The ultimate strength of laminate was evaluated as stress to failure. The sharp decline in strength resulting in a rapid drop in the stress-strain curve was assumed as a criterion of failure.

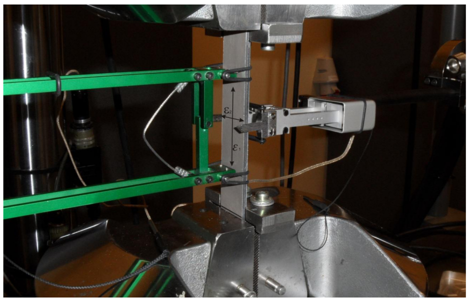

Fig. 2. FML strength properties testing stand

Rys. 2. Stanowisko do badań właściwości wytrzymałościowych laminatów FML

Macrostructural and microstructural characterisations before and after the tensile strength test were carried out using optical microscopes (Nikon MA 200, Nikon SMZ) and scanning microscopes (Zeiss Ultra Plus, NovaNano SEM 450 FEI).

RESULTS AND DISCUSSION

Tensile properties

The tensile characteristics of the metal and fibre metal laminate are characterised by semi-bilinear stress-strain curves, while those of the composites by a linear curve (Fig. 3).

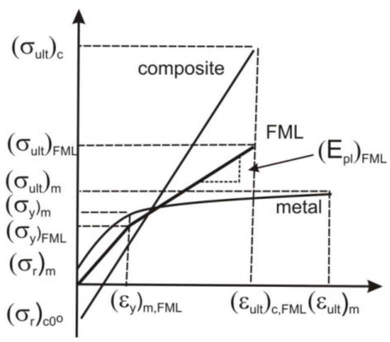

Fig. 3. Schematic uniaxial stress-strain curves for metal, prepreg (composite) and laminate (FML) by [8]; designations in text

Rys. 3. Schematyczne krzywe naprężenie-odkształcenie dla metalu, kompozytu i laminatu (FML) wg [8]; oznaczenia w tekście

The composite reinforced by continuous fibre demonstrates linear elastic properties until failure. The decisive factor affecting tensile strength is fibre elasticity, and, to a lesser extent, the properties of the polymer matrix (yield stress $(\sigma_y)_c \cong$ ultimate strength $(\sigma_{ult})_c$ for elastic strain $(\varepsilon_{ult})_c$). The cured composite is also subject to compressive residual stress $(\sigma_r)_c$. The metal has elastic properties approximately to the limit $(\sigma_y)_m$, with the possible presence of residual stress $(\sigma_r)_m$. The continued increase in stress causes plastic deformation until the tensile strength limit $(\sigma_{ult})_m$ is reached for strain $(\varepsilon_{ult})_m$. An FML should reach a strain level similar to the strain of the composite for stress higher than metal $(\sigma_{ult})_{FML} > (\sigma_{ult})_m$, without cracking of the metal layers.

The stress-strain correlation for an FML in the elastic range is expressed by Equation (1):

$$(\sigma_{el})_{FML} = \frac{E_m d_m + E_{FRP} d_{FRP}}{d_{FML}} \cdot (\varepsilon_{el})_{FML} \tag{1}$$

where: E_m and E_{FRP} represent Young's moduli for the metal and composite respectively, d_m , d_{FRP} , d_{FML} are the total widths of: the metal layers, composite layers and the whole laminate, respectively [4, 5].

The stress-strain correlation for an FML in the plastic range is expressed by Equation (2):

$$(\sigma_{pl})_{FML} = (\sigma_y)_m \cdot \frac{d_m}{d_{FML}} \left(1 - \frac{E_{pl}}{E_{el}}\right)_m + (E_{pl})_{FML} \cdot (\varepsilon_{pl})_{FML} \tag{2}$$

where: E_{pl} is the modulus expressed as the tangent of the angle of slope of the stress-strain curve in the plastic range (Fig. 3) [4, 5].

Investigating the mechanical properties of FMLs by analytical methods on the basis of the classical theory of laminates constitutes a simplified method. In the elastic range a good level of conformity of analytical calculations (Eq. (1)) and experimental results (Table 1, Fig. 4) is achieved, but the calculations fail to take into consideration the presence of the anodised layer. The stress calculations in the plastic range result in significant discrepancies in comparison with the experiment. For example, for the Al/GFRP [0°] laminate, assuming on the basis of the stress-strain curves for the aluminium alloy the value $(E_{pl})_{Al}$ = 17.544 GPa and for the FML the value $(E_{pl})_{FML}$ = 14.3 GPa, the result obtained from Equation (2) is $(\sigma_{ult})_{FML}$ = = 762 MPa, while the tensile strength test demonstrates a tensile strength above 900 MPa (Table 1, Fig. 4a). In consideration of the simplified calculation model that does not take into account the intermediate anodised layer and the metal-composite adhesion force, uniaxial tensile strength tests were conducted and the separation surfaces of the laminates were analysed to determine the degradation mechanism of the laminates during uniaxial tensile strength testing.

The results of the tensile test of the studied materials are listed in Table 1 and presented in Figure 4.

TABLE 1. **Tensile properties of tested fibre metal laminates**
TABELA 1. **Właściwości wytrzymałości na rozciąganie badanych laminatów metalowo-włóknistych**

Material	Fibre direction	Surface treatment ***	Young's modulus E [GPa]	Yield strength σ_y [MPa]		Tensile Ultimate Strength σ_{ult} [MPa]
				experiment	Eq. (1)	
Aluminium / GFRP	0	(Cr+P)	62.87 (±0.57)	323 (±2.65)	322	918.8 (±24.21)
	0/90	(Cr+P)	57.63 (±4.27)	324.33 (±2.31)	296	603.9 (±9.13)
	±45	(Cr+P)	52.26 (±0.71)	252.67 (±2.08)	No data	365.62 (±2.49)
	0	(S+P)	62.52 (±1.55)	326.33 (±4.93)	322	904.43 (±10.20)
	0/90	(S+P)	57.67 (±1.39)	269.33 (±7.88)	296	566 (±17.52)
	±45	(S+P)	52.5 (±0.70)	228 (±5.66)	No data	335.5 (±9.19)
Aluminium 2024- T3[888]	-		73.5	min. 290	-	min. 490
TVR 380 M12/26%/R-glass	0	-	46.4 (±1.21)	-	-	1534 (±55.3)
	90	-	14.9 (±0,04)	-	-	74.5 (±3.5)

*(Cr+P) - anodising in chromic acid + primer
**(S+P) - anodizing in sulphuric acid + primer
*** ASM Metals Reference Book, Third edition, Michael Bauccio, Ed. ASM International, Materials Park, OH, 1993.

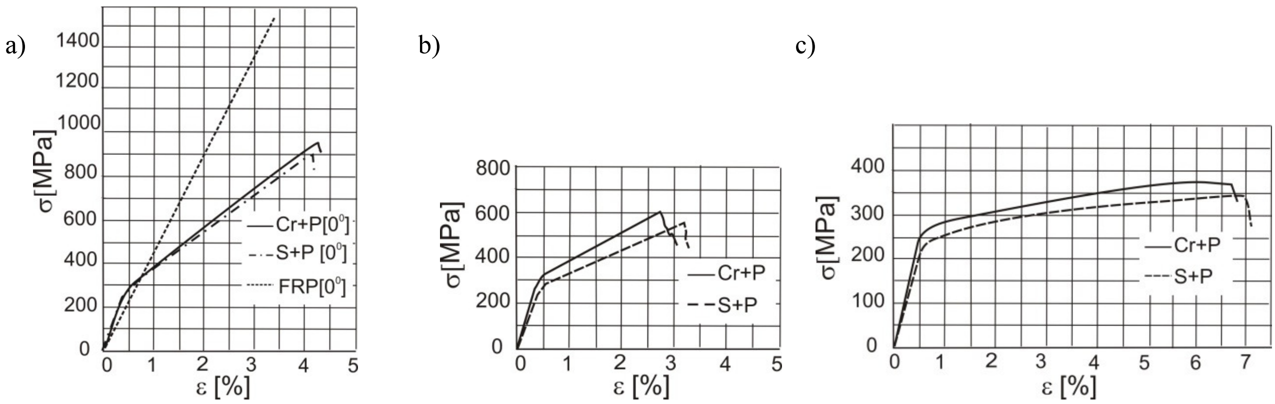

Fig. 4. Typical stress-strain curves for laminates with different fibre orientations and anodising: a) [0°] Cr+P, S+P, b) [0/90°] Cr+P, S+P, c) [±45°] Cr+P, S+P; FRP[0°] - sample of UD glass composite

Rys. 4. Krzywe naprężenie-odkształcenie dla laminatów o różnych kierunkach ułożenia włókien i sposobie anodowania: a) [0°] Cr+P, S+P, b) [0/90°] Cr+P, S+P, c) [±45°] Cr+P, S+P; FRP[0°] - próbka z kompozytu szklanego

The tensile strength of fibre metal laminates depends mainly on the orientation of the reinforcing fibres in the composite layers (Fig. 4, Table 1). In the case of configuration [±45°], the slope of the curve in the plastic deformation range is lower than for other configurations and the relative deformation is higher. In laminates with a [0°] fibre arrangement in relation to the tension direction, the tensile strength is the highest and significantly higher in comparison to the applied metal material. Laminates with a [0/90°] configuration are characterised by an intermediate tensile strength limit and the lowest strain.

The strength is less dependent on the method of metal surface preparation but the values obtained for specimens anodised in chromic acid were higher than in sulphuric acid (Fig. 4, Table 1).

Examination of fractured surfaces

Owing to the fact that the specimens were not broken, the fragments of metal sheet/ composite were termed "fractured" and subjected to detailed macroscopic and microscopic observations. Figure 5 shows an example of a macroscopic view of samples with two types of anodising layers after tensile strength testing.

a)
b)

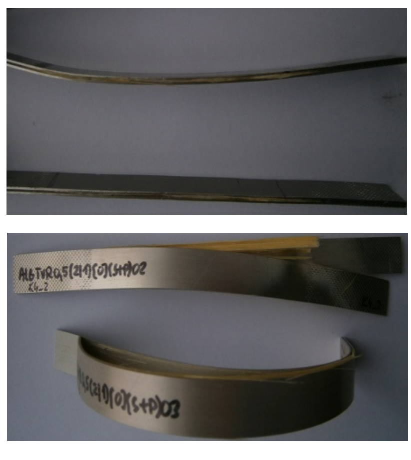

Fig. 5. FML laminate samples after tensile strength test: a) anodising in Cr+P, b) anodising in S+P; [0°]

Rys. 5. Próbki laminatu metalowo-włóknistego po testach na rozciąganie: a) anodowanie Cr+P, b) anodowanie S+P; układ [0°]

The nature of destruction is significantly diversified, particularly depending on the aluminium surface preparation (Fig. 5). The higher deformation level of the sheet anodised in sulphuric acid is caused by higher stress levels on the metal/anodised layer/composite boundary. The oxide layer produced in the SAA process is less porous (Fig. 6), thicker and harder than that produced in CAA, with lower adhesion to the composite, which was demonstrated in earlier tests [9]. This leads to a difference in the studied static mechanical properties of the laminate.

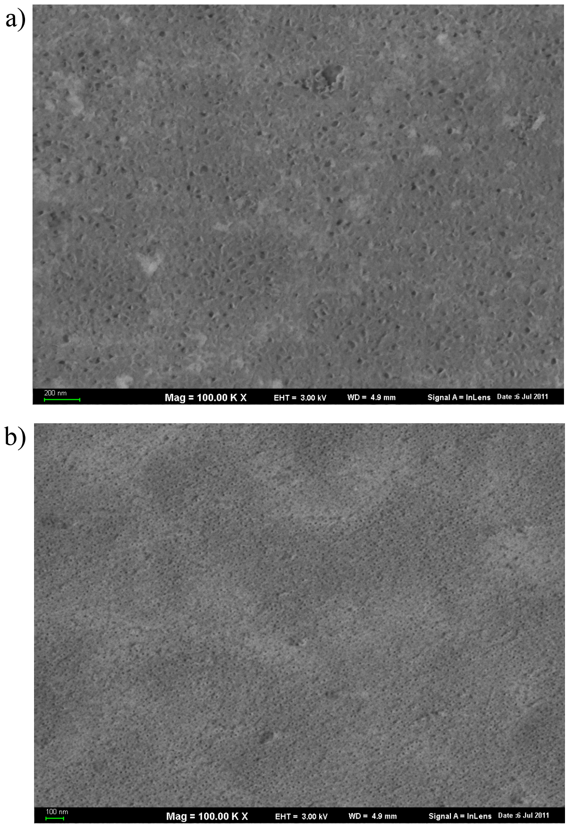

Fig. 6. Anodic oxide layer on aluminium alloy: a) CAA, b) SAA; SEM

Rys. 6. Warstwa anodowa na stopie aluminium: a) CAA, b) SAA; SEM

The energy stored during the test is released in the form of fibre fracture toughness, composite decohesion, and composite/metal delamination, which results in aluminium sheet strain.

Macroscopic observations of the delaminated samples demonstrate that some fibres which partly bonded with the polymer matrix remain on the sheet surface, while their amount depends on the lay-up direction of the layers (Figs. 7b,d, 9-11). Figures 7a and b illustrate specimens containing aluminium anodised in chromic acid with the fibre composite in the [0°] direction. In the course of the tensile strength test, the metal sheets were not broken nor cracked but partial decohesion occurred in the composite layer. The same macroscopic destructions were observed for the [±45°] fibre layout (Fig. 7c,d). However, Figure 5b illustrates the specimens anodised in sulphuric acid and representing complete structure degradation in the polymer composite as well as delamination on the aluminium - polymer layer interface. Moreover the aluminium sheets did not break. The presence of composite fragments on the metal surface for both anodising types indicates that the metal / composite adhesion is higher than the resin / fibre adhesion.

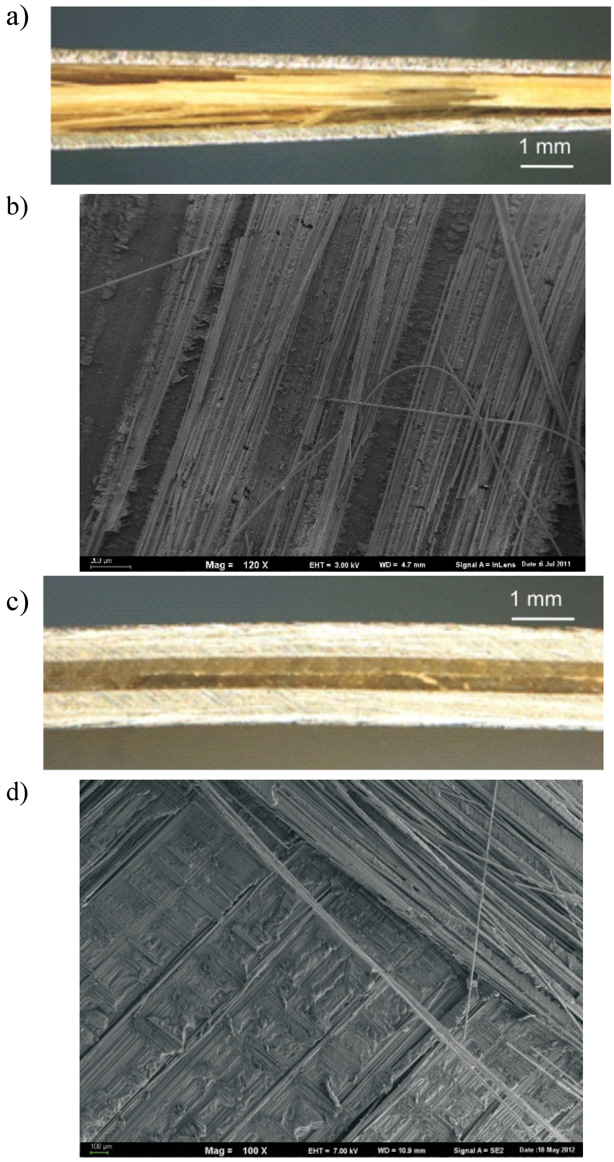

Fig. 7. Degradation of FML sample structure after tensile strength test: a) macrostructure of Al/GFRP[0°], b) surface of sheet with residual of [0°]composite, c) macrostructure of Al/GFRP[±45°], d) surface of sheet with residual of [±45°] composite; Cr+P anodizing

Rys. 7. Zniszczenie struktury próbki FML po teście rozciągania: a) makrostruktura Al/GFRP[0°], b) powierzchnia blachy z pozostałością kompozytu [0°], c) makrostruktura Al/GFRP[±45°], d) powierzchnia blachy z pozostałością kompozytu [±45°]; anodowanie Cr+P

Figures 8-11 present the microstructure of the "fracture" after strength testing. The description of the composite layer degradation in FMLs uses terms featured in the literature on fibre-polymer composites [10, 11].

Lateral cracks can be noticed in the fibres as well as in the anodised layer (Fig. 8).

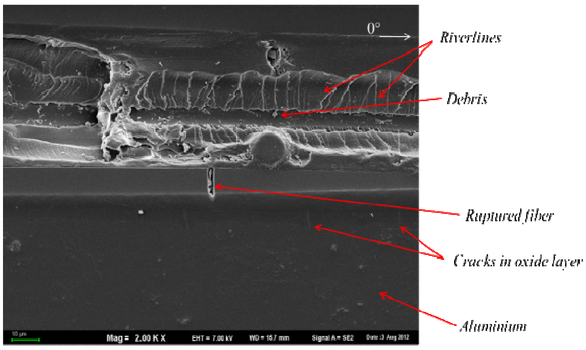

Fig. 8. Cracks and degradation of FML interface, direction [0°], longitudinal section, anodising Cr+P, SEM

Rys. 8. Pęknięcia i degradacja powierzchni rozdziału w FML, kierunek 0°, przekrój wzdłużny, anodowanie Cr+P, SEM

a)

b)

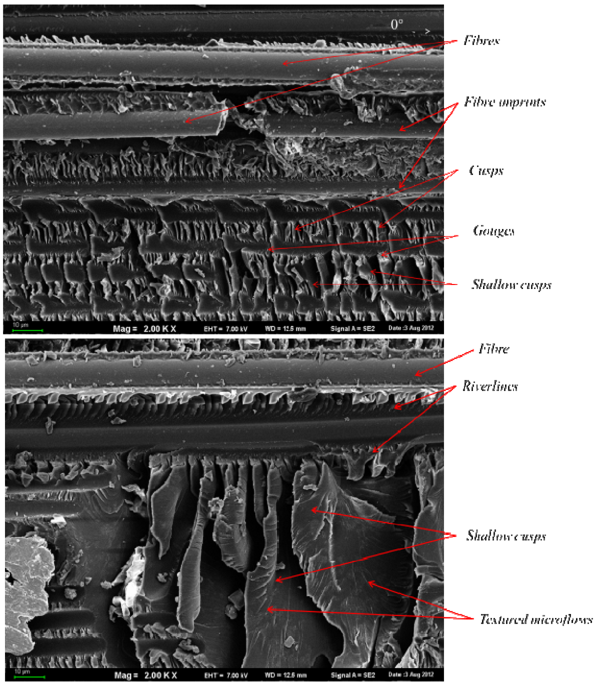

Fig. 9. Aluminium surface morphology after tension test, fibre configuration [0°], anodizing Cr+P; SEM

Rys. 9. Morfologia powierzchni aluminium po testach rozciągania, układ włókien [0°], anodowanie Cr+P; SEM

The degradation called *riverlines* and *debris* (Fig. 8, 9b) occurs in the matrix area. The *rolling* and flowing of resin - specific for tension phases - are revealed under high magnification in the form of *textured micro-flows*, *gouges, cusps and shallow cusps* (Fig. 9). Broken fibres are present in the resin adhering to the metal surface, while traces of separated fibres are also visible as *fibre imprints* (Figure 9a).

Total degradation of the composite layer structure and partial of the metal/composite interface was observed in the [0/90°] fibre arrangement configuration. Delamination occurs between the [0°] and [90°] layers combined with displacement of the overlapping [90°] layer as illustrated in Fig. 10a. The cracks occurring in the anodised layer are combined with lateral cracks in the matrix (Fig. 10b).

a)

b)

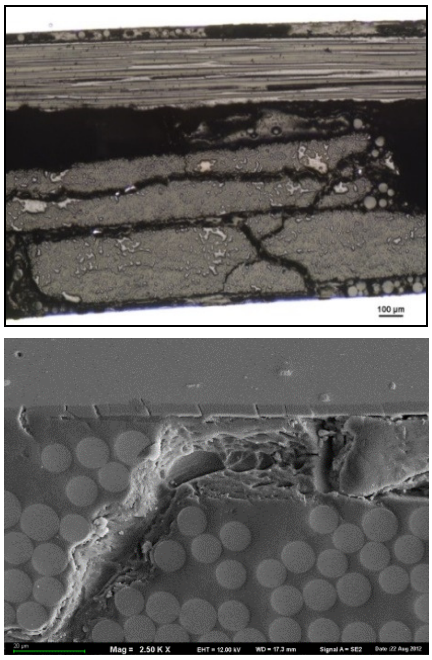

Fig. 10. Degradation of laminate structure with [0/90°] configuration: a) optical microscope, b) SEM; Cr+P anodising

Rys. 10. Degradacja struktury laminatu z konfiguracją [0/90°]: a) mikroskop optyczny, b) SEM; anodowanie Cr+P

a)

b)

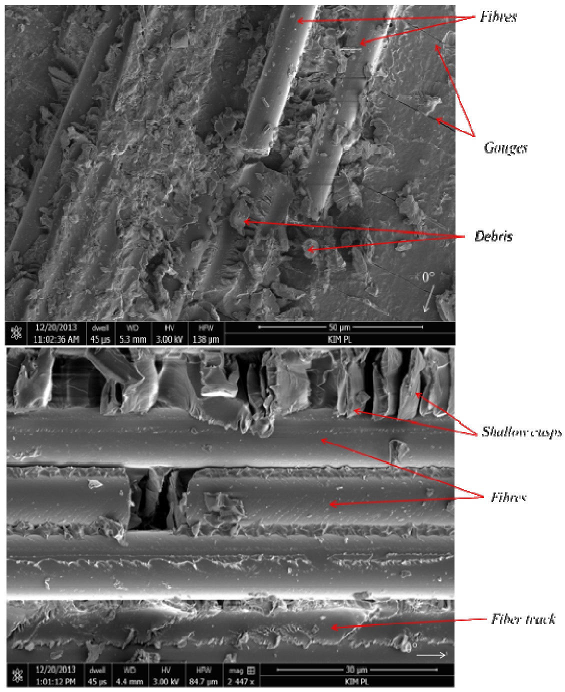

Fig. 11. Aluminium surface morphology after tension tests, S+P anodising, fibre layout [0°]; SEM

Rys. 11. Morfologia powierzchni aluminium po testach rozciągania, anodowanie S+P, układ włókien [0°]; SEM

The laminate with aluminium anodised in sulphuric acid was characterised by complete structural delamination on the aluminium/composite interface with a decohesion mechanism (with residual presence of the composite on the metal surface). The image of the residual presence of the composite on the metal surface does not differ from the image observed in the case of metal anodised in chromic acid, i.e. *gouges, debris, shallow cusps and fibre tracks* are present. Figure 11 illustrates the "fracture" morphology after the tensile strength test on such specimens. There are visible fibres, separations between the resin and fibres, and broken fibres. The lateral cracks are oriented laterally to the fibre arrangement direction.

An analysis of the microstructures of the laminate with various fibre arrangement directions primarily demonstrated the phenomenon of internal degradation of the polymer composite structure, regardless of the surface treatment. All the samples showed partial delamination between the anodised aluminium and the epoxy/glass composite.

CONCLUSIONS

The following conclusions were drawn on the basis of the conducted experiments:

- The tensile strength of the studied laminates was expressed as the stress level at which decohesion of the composite without cracking in the aluminium sheet occurs.
- Residual stress causes deformation and delamination in the laminates after the uniaxial tensile test, especially after the application of SAA.
- The fibre arrangement direction is a decisive factor determining the characteristics of the stress-strain curves, while the type of anodised layer is significantly less important.
- The presence of epoxy resin and fragments of fibres on the sheet surface after the tensile strength test demonstrates that the metal/composite adhesion force is higher than the composite cohesive force.
- The anodising method, with high composite/metal adhesion, does not affect the degradation mechanism of the laminates in the tensile strength test. Cracking of the anodised layer, transverse cracking of the fibres and propagation of the failure depending on the lay-up direction of the prepreg layers in relation to the direction of the applied force are observed.
- The surface morphology of the failure area caused by the decohesion of the composite in an FML is of the same nature as the fracture in the composite material.

Acknowledgements

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